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Indian Standard

PESTICIDE - METHOD FOR DETERMINATION OF RESIDUES IN AGRICULTURAL AND FOOD COMMODITIES, SOIL AND WATER — CAPTAFOL

UDC 664 : 543 [632’95’028 CAP]

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MA-RG
NEW DELHI 110002

August 1993

Price Group 2

Pesticides Residue Analysis Sectional Committee, FAD 34

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Residue Analysis Sectional Committee had been approved by the Food and Agriculture Division Council.

Captafol [cis-N (1, 1, 2, 2-tetrachloroethyl thio)-4-cyclohexene-1, 2-dicarboximide] is used in agriculture for the control of fungal diseases. Assessment of its residues in food commodities is therefore an important step in safeguarding human health.

This standard will enable the health authorities and others engaged in the field to follow uniform test procedures for the estimation of captafol residues in food commodities.

In the preparation of this standard, due consideration has been given to the maximum limits of dithiocarbamate residues laid under the provisions of ***Prevention of Food Adulteration Act, 1954*** and the Rules framed thereunder. The test method is restricted to the prescribed level of residues.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance, with IS 2 :1960 'Rules for rounding off numerical values (***revised***)'.

Indian Standard

(Reaffirmed 1998)

PESTICIDE — METHOD FOR DETERMINATION OF RESIDUES IN AGRICULTURAL AND FOOD COMMODITIES, SOIL AND WATER — CAPTAFOL

1 SCOPE

1.1 This standard prescribes the gas chromatographic (GLC) method for determination of captafol residues in agriculture and food commodities, soil and water.

1.2 The method is applicable with a limit of detection in the range of 0’02 µg/g.

1.3 Though no set procedure for thin layer chromatography (TLC) is being prescribed, standardized TLC procedures may be followed, if necessary for the purpose of clean up, identification and confirmation of residues of captafol.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard.

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision)*
11380 : 1985	Method of sampling for the determination of pesticide residues in agricultural and food commodities

3 PRINCIPLE

The captafol residue extracted from the sample dissolved in hexane and estimated gas chromatographically in an instrument equipped with electron capture detector. The content of captafol is determined by comparing the response with the response of a known captafol standard of similar concentration.

4 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (see IS 1070 : 1992) shall be employed in the tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the result of analysis.

5 SAMPLING

The representative samples for the purpose of estimating residues of captafol in food commodities shall be drawn in accordance with IS : 11380 : 1985.

6 APPARATUS

6.1 Waring Blender

6.2 **Vacuum Rotary Evaporator**

6.3 **Chromatographic column** — 25 cm long × 2 cm i.d.

6.4 **Buchner Funnel**

6.5 **Gas Chromatograph**

Equipped with an election capture detector and operating under the following suggested parameters. These parameters may be varied according to the available facilities, provided standardization is done:

Column	: Glass, 100 cm × 0'4 cm, packed with QF-1 on chromosorb G (60-80) mesh	
Temperatures	: Column oven	210°C
	Injector	240°C
	Detector	250°C
Carrier gas (Nitrogen) flow rate	: 40 ml/min	
Retention time	: 2’4 minutes	

6.5 Microlitre Syringe — 10 µl capacity.

7 REAGENTS

7.1 Acetonitrile — GLC grade.

7.2 Acetone — GLC grade.

7.3 Benzene — GLC grade.

7.4 Florisil or Equivalent — Chromatographic Grade

Use as a 3 : 2 (*m/m*) mixture of 100-200 mesh and 60-100 mesh.

7.5 **Celite Filter Aid or Equivalent**

7.6 Hexane

7.7 **Sodium Sulphate** — anhydrous

7.8 **Aqueous Sodium Chloride Solution** — **5** percent (m/v.)

7.9 Captafol Reference Standard — of known purity.

8 EXTRACTION

8.1 General

The method of extraction is common for most food commodities and the procedure described in 7.2 can be used for grains, straw, fruits and vegetables, oils and nuts, as well as meat and egg. However, for high fat substances, the additional acetonitrile partition clean up is required before estimation.

8.2 Grain, Straw, Fruits, Vegetables, Oils, Oilseeds, Nuts, Meat, Egg and Others

Transfer 100 g of finely ground chopped sample into a waring blender, add 50 g anhydrous sodium sulphate followed by 400 ml benzene. Blend the mixture for 15 minutes. Filter the organic phase through fluted filter paper containing a layer of anhydrous sodium sulphate. If emulsions are formed in the mixture, break these by adding 5 g of celite filter aid or equivalent. Re-extract the residue with fresh 200 ml benzene and filter as before. Combine the filtrates and evaporate the solvent to dryness in a vacuum evaporator.

8.3 Soil

Transfer 50 g of air dried and sieved soil into a 500-ml conical flask. Add about 25 g anhydrous sodium sulphate followed by 200 ml benzene. Shake the contents on a rotary shaker for about one hour. Filter the organic phase through Buchner funnel under suction. Wash the Buchner funnel with 20 ml additional benzene. Collect the filtrates and evaporate off the solvent to dryness in a vacuum rotary evaporator.

8.4 Water

Transfer 500 ml of the water sample into a 1 OOO-ml separatory funnel. Add 100 ml of aqueous sodium chloride solution followed by 100 ml benzene to the separatory funnel. Shake the contents well for about 2 minutes and allow the layer of anhydrous sodium sulphate mounted on a funnel. Repeat the extraction twice with 75 ml portions of benzene. Wash the sodium sulphate layer with 10 ml additional benzene. Collect the benzene extracts and evaporate off to dryness in a vacuum rotary evaporator. No additional clean up is required for water samples.

9 CLEAN UP

9.1 General

While the florisil column clean up is recommended for use on all commodities in case of high fat substances like nuts, oilseeds the acetonitrile partition step shall proceed the column clean up.

9.2 Acetonitrile Partition (for High-fat Substances)

Transfer the residue after extract (see 7.2) with three 10-ml portions of hexane to a 100-ml separatory funnel followed by three 10 ml rinses with acetonitrile. Shake well for one minute and allow the phases to separate. Drain off the acetonitrile phase and rinse the hexane phase with 10 ml of fresh acetonitrile. Collect the acetonitrile extracts and evaporate to dryness in a vacuum rotary evaporator.

9.3 Column Clean Up

9.3.1 *Preparation of Chromatographic Column*

Place a glass wool plug in the bottom of the column, add 50 ml hexane and 15 g florisil or equivalent. Rinse the sides of the column with hexane and cover the florisil with a 15 g layer of anhydrous sodium sulphate. Allow the hexane to drain to the top of the column packing.

9.3.2 *Clean Up*

Transfer the residue after extraction (see 7.2) or after acetonitrile partition (see 8.2) as the case may be, with three 10 ml portions of hexane to the column and allow the sample to drain to the top of the column packing. Wash the column with 50 ml hexane, followed by 50 ml benzene and discard the washings. Fmally elute captafol with 250 ml benzene. Evaporate the elute to dryness in a vacuum evaporator.

10 PROCEDURE

10.1 Preparation of Standard Solution

Prepare solution of captafol reference standard (*see* 6.9) in hexane with concentration of 0'01 to 10 $\mu g/ml$.

10.2 Preparation of Sample Solution

Dissolve the residue after clean up (see 8.3) in 10 ml of hexane.

10.3 Estimation

Inject simultaneously 2'0 μl of standard and sample solutions into the gas chromatograph. Identify the peaks by the retention time and measure the peak areas of standard and sample.

11 CALCULATION

Residue of Captafol ($\mu g/g$)

$$= \frac{A_1 \times V_2 \times V_3 \times C}{A_2 \times V_1 \times M} \times f$$

where

A_1 = peak area of the sample;

V_2 = volume, in μl, of standard captafol injected;

V_3 = total volume, in ml, of the sample solution;

C = concentration, in µg/g, of the standard solutions;

f = recovery factor = $\dfrac{100}{\text{percent mean recovery}}$

A_2 = peak area of the standard;

V_1 = volume, in µl, of the sample injected; and

M = mass, in g, of the sample taken for analysis.

NOTE — Percent mean recovery is determined by taking untreated control sample to which a known amount of captafol is added and **analysed** as described above.

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